

Mechanical Properties of Poly(ether ether ketone)/Poly(ether ketone) Blends: Use of Simple Models Relating Normalized Tensile Parameters

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Received 10 February 2009; accepted 2 August 2009

DOI 10.1002/app.31353

Published online 26 March 2010 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: In this study, mechanical properties such as tensile properties, flexural properties, and Izod impact strength of poly(ether ether ketone) (PEEK) and poly(ether ketone) (PEK) blends at PEK concentration from 0 to 0.42 volume fraction were studied. The blends of PEEK and PEK of different compositions were prepared by extrusion in a single-screw extruder. With increase in the PEK concentrations, the tensile strength, flexural strength, and modulus increased whereas the tensile modulus and the impact strength decreased. Homogeneous dispersion and

adhesion of PEK in PEEK was shown by the morphological studies. Crystallinity of blends influenced the tensile modulus and the impact strength. Using simple models to relate normalized tensile parameters where the data were divided by the crystallinity of the blends and of the PEEK matrix, respectively, supported the experimental results. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 117: 849–856, 2010

Key words: PEEK/PEK blends; mechanical properties; impact strength; morphology; normalized models

INTRODUCTION

The widespread use of polymeric materials with good mechanical properties, high service temperature, and improved chemical resistance has led to active research in the development of new high performance engineering polymer materials.^{1–4} Aromatic polymers have drawn much attention and gained wide practical uses because of their excellent thermal and chemical stability, mechanical properties, and radiation resistance.^{5,6} These polymers are of interest for various applications in areas with a high radiation field, such as in aerospace systems and around fusion reactors.⁷ Poly(ether ether ketone) (PEEK) is a semicrystalline engineering polymer which has attracted a lot of attentions in the recent past because of its excellent thermal and mechanical properties.^{8–10} PEEK is a new generation engineering polymer with remarkable properties, which are still under-explored. There are problems related to all the engineering resins that is of initial wastages both in terms of loss of time and raw material during stabilization of the process. There is expectation of excellent mechanical properties in combination with ease of processing along with long-term durability (i.e., excellent wear¹¹ and thermal properties) for getting

intricate shapes, designs, and better surface finish. PEEK nearly meets all the requirements but still there is lot of scope for improvement. Poly(ether ketone) (PEK) is also a semicrystalline material with good chemical resistance, high heat resistance, good wear resistance, and high strength.¹² According to Utracki,¹ since the maximum extent of crystallinity (determined by chain mobility) is about 50%, during cooling the isomorphous blend with high content of higher melting ingredient crystallizes first, leading to a substantial difference in composition of the amorphous and crystalline after solidification. PEEK and PEK are high performance semicrystalline thermoplastic¹³ and belong to the same poly(aryl ether ketone) family. Since the two polymers differ only in the presence of an extra ether linkage, so it was expected that the blends of these two polymers would exhibit isomorphous behavior.^{9,13–15} In a previous study,¹⁴ it was shown that solution blends of PEEK and PEK cocrystallize on rapid quenching from the melt but no detail study regarding morphology and mechanical properties has been carried out. In this study, blends of PEEK and PEK have been prepared by extrusion and have been characterized for their various properties. The objective of the present investigation is to study the effect of PEK on the mechanical properties of PEEK. The blends containing varying proportions of the PEK were characterized by the evaluation of tensile, flexural, and impact properties. Tensile properties were compared with simple theoretical predictions.

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TABLE 1
Properties of Pure PEEK and Pure PEK

Properties	PEEK	PEK
Molecular weight	34700	23600
Density (amorphous), g/cm ³	1.382	1.430
Density (crystalline), g/cm ³	1.267	1.272
Tensile strength, MPa	92.0	104.0
Tensile modulus, GPa	3.56	3.19
Elongation at break	25.4	30.2
Impact strength Izod, Jm ⁻¹	84	59
Flexural strength, MPa	170	175
Flexural modulus, GPa	3.89	3.98

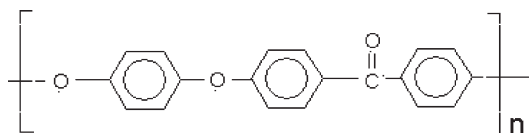
All the data are taken from the data sheet.

EXPERIMENTAL

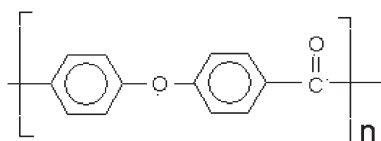
Materials

The materials used in this study were PEEK (Victrex 450G) and PEK (P-22) from ICI Co. PEEK is a semi-crystalline polymer with glass transition temperature of 143°C and melting point of 345°C.¹³ PEK is also semicrystalline polymer with glass transition temperature of 155°C and melting point of 365°C.¹³ Properties of both pure PEEK and pure PEK are given in Table I.

The structures of polymers are:



PEEK



PEK

Blending

Polymers were preheated at 120°C for 2 h in an air circulating oven before blending to remove moisture. Because during processing the moisture could be entrapped inside the material and as a result the material would show lower mechanical properties. PEEK was blended with varying proportions of the PEK (0.052–0.42, volume fraction, Φ_d) by first mixing physically and then compounded in HAAKE Rheocord 9000, a laboratory size single-screw extruder. The processing conditions of all the compositions were as follows: feed zone (Z_1) temperature 365°C, compression zone (Z_2) temperature 380°C, metering zone (Z_3) temperature 395°C, die zone (Z_4) temperature 385°C,

screw speed 30–35 rpm, torque 18–25 Nm, and cutter speed 110 m/min. The molten extrudate was quickly quenched in a water bath to room temperature. The blends, thus obtained were collected as strands and cut into granules using an automatic chopper. The chopped granules were dried at 120°C for 5 h and molded into sheets of thickness 3 mm by compression molding for further characterization.

Characterization

Tensile and flexural properties were determined by a universal testing machine (Zwick 1773) at room temperature. Samples were cut into standard tensile (ASTM D638) and flexural (ASTM D790) bar. The crosshead separation was 6 cm and crosshead speed was 20 mm/min during the tensile test following the ASTM D638 test method.¹⁶ The test parameters employed for flexural properties were as follows: span-to-depth ratio = 32 : 1, crosshead speed = 5 mm/min following the ASTM D790.¹⁶ Notched bar specimens were used for the evaluation of the Izod impact strength on a Dynatop drop weight system according to the ASTM D256 test procedure.¹⁶ A minimum of five samples were tested for each composition and their average value within $\pm 3.5\%$ is reported. The tests were performed at ambient temperature 303 ± 2 K. Fractured surface of specimens obtained during flexural test were observed in a scanning electron microscope (CARL ZEISS EVO-50) to study the morphology of the blend samples. The samples were coated with a thin layer of gold by using a vacuum sputtered prior to SEM observations. Differential scanning calorimetry (DSC) were undertaken to measure the extent of crystallization in PEEK and the blends following procedures described elsewhere.¹⁷ For estimation of crystallinity (%) of PEEK in the blends by the DSC method the enthalpy value used for 100% crystalline PEEK was 130 J/g.¹⁸

RESULTS AND DISCUSSIONS

Degree of crystallinity

The crystallinity of PEEK matrix decreased continuously in the presence of increasing contents of the PEK, Table II, except at $\Phi_d = 0.25$. At this particular

TABLE II
Crystallinity (%) of PEEK/PEK Blends by DSC Method

Φ_d	Crystallinity (%) (DSC method)
0	33.7
0.05	33.4
0.11	33.2
0.18	32.6
0.25	33.1
0.33	32.5
0.42	32.3

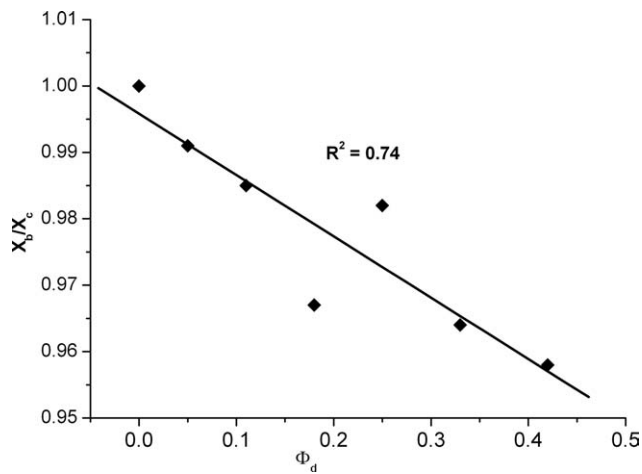


Figure 1 Variations of normalized crystallinity of PEEK/PEK blends (\blacklozenge) (X_b/X_m) against Φ_d .

Φ_d , a kind of nucleation for crystallization may be favored in the presence of PEK as was also reported in literature.¹³ The crystallinity data were determined by the DSC. In the analysis of the properties of the PEEK/PEK blends, the crystallinity values by the DSC method were used. The normalized crystallinity data [ratio of the crystallinity of PEEK in the blend, X_b , to that of the PEEK matrix, X_m] decreased linearly with the increasing volume fraction (Φ_d) of the PEK with a correlation coefficient (R^2 value) of 0.74 (Figure 1).

Tensile properties

The tensile properties were first determined from the primary stress-strain curves of the blends (not shown). In Figures 2–7, the results are presented as

F2–F7

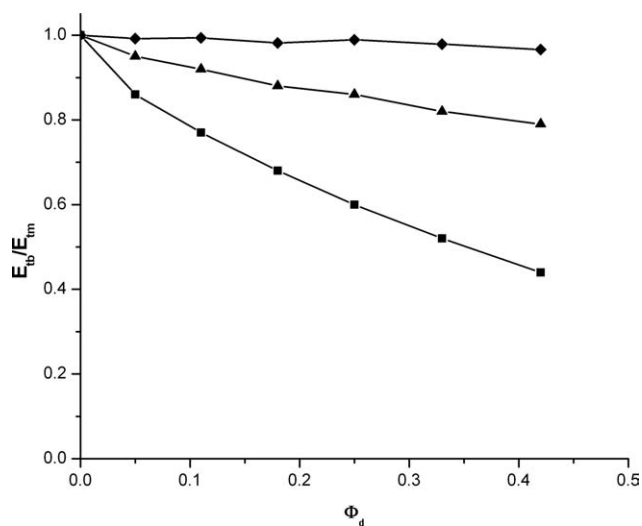


Figure 2 Plot of relative tensile modulus (E_{ib}/E_{tm}) of PEEK/PEK blends (\blacktriangle) and the predictive models according to the "Rule of Mixtures" (\blacklozenge) [eq. (1)] and "Foam Model" (\blacksquare) [eq. (2)] against Φ_d .

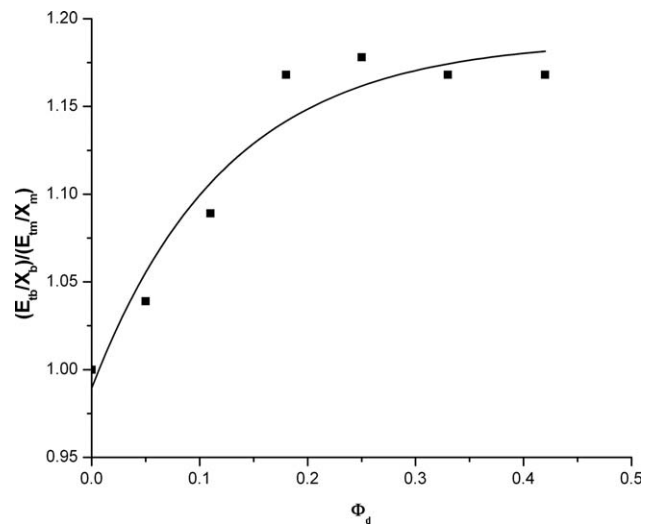


Figure 3 Dependence of normalized relative tensile moduli [$(E_{ib}/X_b)/(E_{tm}/X_m)$] of PEEK/PEK blends (\blacklozenge) as a function of Φ_d .

the ratio of the property of the blends (subscript b) to that of the PEEK matrix (subscript m) versus Φ_d of the PEK. Some of the data are presented in the normalized form by dividing the moduli and strength with the crystallinity of the PEEK in the blend and the matrix and plotted as functions of Φ_d .

Tensile modulus

Figure 2 shows the plot of relative tensile modulus (E_{ib}/E_{tm}) of the PBT/FE blends against Φ_d . The modulus decreased with increase in Φ_d , at the highest Φ_d the modulus decreased by $\sim 18\%$ from that of the matrix. This indicates that PEEK is softened to an extent by the PEK.

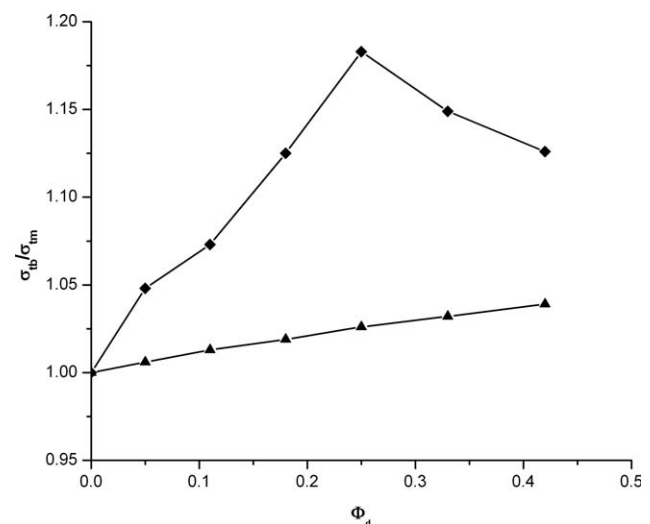


Figure 4 Plot of relative tensile strength (σ_{ib}/σ_{tm}) of PEEK/PEK blends (\blacklozenge) and the predictive models according to the "Rule of Mixtures" (\blacktriangle) against Φ_d .

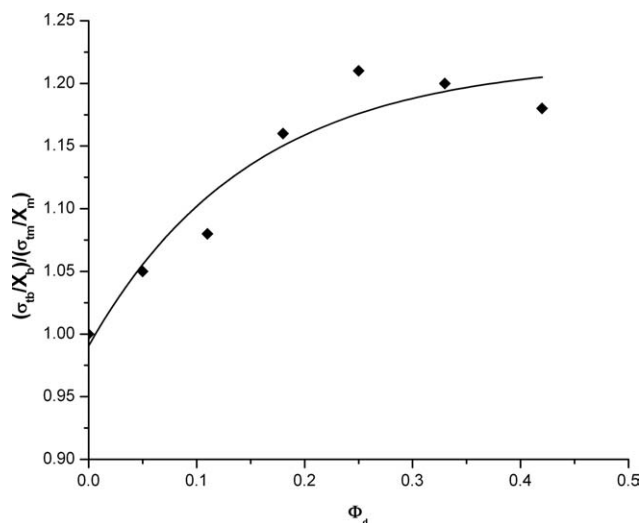


Figure 5 Dependence of normalized relative tensile strength $[(\sigma_{tb}/X_b)/(\sigma_{tm}/X_m)]$ of PEEK/PEK blends (◆) as a function of Φ_d .

To understand the role of PEK in the blend structure the data were analyzed according to simple predictive model following the “rule of mixture”^{19,20} as in composites and blends, eq. (1), and the “foam model” of Cohen and Ishai,²¹ eq. (2):

$$E_{tb}/E_{tm} = [E_{td}/E_{tm} - 1]\Phi_d + 1 \quad (1)$$

$$E_{tb}/E_{tm} = (1 - \Phi_d^{2/3}) \quad (2)$$

The moduli values of the PEEK (E_{tm}), PEK (E_{td}), and the blends (E_{tb}) were determined experimentally. The “rule of mixture” exhibited reasonably good agreement with the data whereas the “foam model” curve showed lower values. This indicates

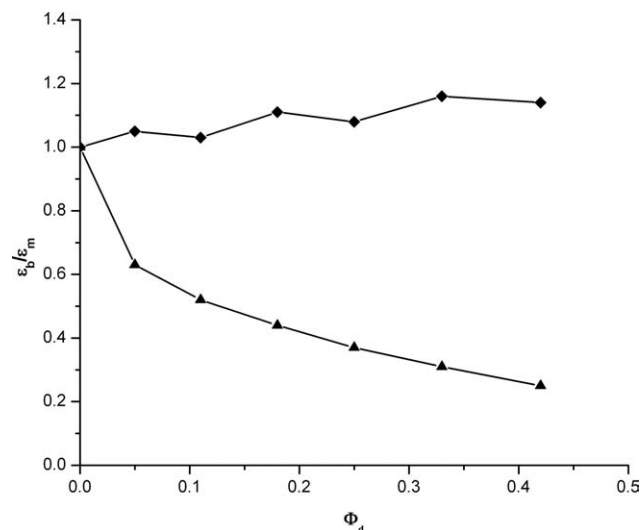


Figure 6 Plot of relative elongation-at-break (ϵ_b/ϵ_m) of PEEK/PEK blends (◆), and Nielsen's model (▲) [eq. (6)] against Φ_d .

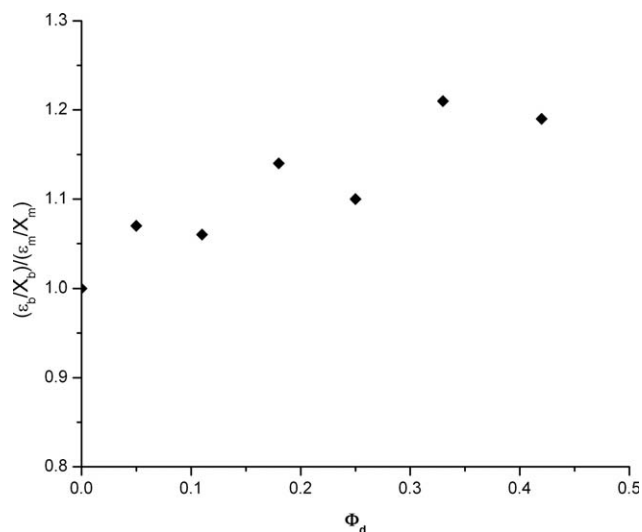


Figure 7 Variations of normalized relative elongation-at-break $[(\epsilon_b/X_b)/(\epsilon_m/X_m)]$ of PEEK/PEK blends (◆), as a function of Φ_d .

that the dispersed phase decreases the stiffness of the PEEK matrix. This is, however, quite expected as the modulus of the PEK is lower than that of the PEEK matrix. The crystallinity of the blends also decreases as a result of the interaction (Table I), which will tend to decrease the modulus. Since the crystallinity of PEEK decreased in the presence of PEK it may indicate some kind of interphase interaction. To evaluate the effect of this phase interaction the relative moduli data were normalized by dividing the moduli with the crystallinity of the PEEK in the blend and the matrix $[(E_{tb}/X_b)/(E_{tm}/X_m)]$ and plotted as functions of Φ_d (Figure 3). The data were higher than unity increasing slowly up to $\Phi_d = 0.25$, which then leveled off with further increase in Φ_d . This implies that there is an extent of phase interaction of the PEK with PEEK. This happened because of the influence of overall crystallinity and crystalline shape in addition to the degree of compatibility of the blend system.¹³ The overall crystallinity can be explained on the basis of kinetic arguments. During the blending, as the melt is cooled from above the melting point of higher melting constituent, that is, PEK to below its T_m , first the higher melting constituent or PEK will start crystallizing. At this stage, the lower melting constituent is still in the melt state. As crystallization process proceeds further, crystalline phase will be having more concentration of the component which is kinetically favored, that is, PEK. As the melt is further cooled and crystallization process continues, the concentration of PEK that can enter into the crystalline phase depletes and this favors the other crystallizing polymer in the melt, that is, PEEK to enter into the crystalline phase. But till then most of the surface will be covered by the PEK crystalline phase. This process will continue till

whole of the surface gets covered with spherulites and spherulitic impingement prevents further crystallization. Therefore, further crystallization is not possible and at this stage blend contains more PEEK than PEK as crystalline phase of PEK is covered by crystalline phase of PEEK. So, higher amount of PEK in the blend does not favor this kinetics. As a result, normalized tensile strength and modulus initially increased then leveled off.

Tensile strength

Figure 4 shows the plots of relative tensile strength, that is, ratio of tensile strength of PEEK/PEK blends to that of PEEK, σ_{tb}/σ_{tm} , versus Φ_d . The tensile strength showed a continuous increase with increasing Φ_d indicating that the blend structure was strengthened by the PEK.

Predictive models were used to analyze the tensile strength data of polymer blends to assess the level of interfacial interaction. Three models were used to analyze the tensile strength results obtained in this study are as follows:

Model 1

Nielsen's first power law model²²

$$\sigma_{tb}/\sigma_{tm} = (1 - \Phi_d)S \quad (3)$$

Model 2

Nielsen's two-third power law model²²

$$\sigma_{tb}/\sigma_{tm} = (1 - \Phi_d^{2/3})S^0 \quad (4)$$

Model 3

Nicolais and Narkis Model²³

$$\sigma_{tb}/\sigma_{tm} = (1 - K_b\Phi_d^{2/3}) \quad (5)$$

where σ_{tb} and σ_{tm} represent the tensile strength of the blend and the PEEK, respectively. Φ_d is the volume fraction of PEK in the blends, S and S^0 are the Nielsen's parameter in the first and two-third power law models, respectively. These parameters account for the weakness in the structure brought about by the discontinuity in stress transfer and generation of the stress concentration at the interfaces in case of composites and blends. The value of S and S^0 is unity for no stress concentration effect. K_b in eq. (5) is an adhesion parameter; the maximum value of K_b being 1.21 for spherical inclusion of the minor phase having no adhesion.²³ The three models described earlier have been employed to analyze the tensile strength results to evaluate interfacial adhesion, if any, by comparing the experimental values with those predicted by the models. The values of S , S^0 , and K_b are listed in Table III giving a comparison

TABLE III
Values of Stress Concentration and Adhesion Parameters (S , S^0 , and K_b) in PEEK/PEK Blends

Φ_d	S	S^0	K_b
0	–	–	–
0.05	1.11	1.22	–0.34
0.11	1.21	1.39	–0.32
0.18	1.37	1.65	–0.39
0.25	1.58	1.96	–0.46
0.33	1.71	2.20	–0.31
0.42	1.92	2.53	–0.20
Mean	1.48	1.83	–0.34

between the experimental data and theoretical models.

It was found from the aforementioned models that the experimental values of the blends are higher than those predicted from the aforementioned models taking into account the values of $S = 1.00$ and 1.48, respectively. The relative tensile strength of all the compositions predicted from the model 1 with $S = 1.00$ is found to be 0.78, while the value is found to be 1.15 with $S = 1.48$.

The experimental relative tensile strength values of the blends are found to be higher when compared with the values predicted from model 1. This shows that all the blend compositions can take excess stress since the blend is miscible.¹³ Similarly, the experimental relative tensile strength values are found to be higher when compared with the values predicted from model 2 taking into account $S^0 = 1.00$ and 1.83. This is again in favor of higher interfacial adhesion of the blends. Thus, by comparing the values of Nielsen's parameters (S and S^0) of the two power laws, it is found that the extent of deviation of S value from 1.00 is less than that of S^0 value. This shows that the first power law establishes its better suitability than the fractional power law model.

In the Nicolais–Narkis model [eq. (5)], the area fraction of the inclusion is considered effective.^{23,24} The value of the phase interaction parameter, also known as the weightage factor, K_b , describes the blend structure. For hexagonal packing of the dispersed phase in the plane of highest density $K_b = 1.1$. With no adhesion in the presence of spherical dispersed phase, $K_b = 1.21$.^{23–26} $K_b = 1$ stands for no stress concentration²² and when the dispersed phase does not weaken the structure, $K_b = 0$. Values of $K_b < 1.21$ indicate interphase adhesion, the lesser the value the better the adhesion.^{17,27,28} The analysis also shows an average value of K_b to be –0.34 that is much less than 1.21. The interesting fact is that, the parameter K_b , in the Nicolais–Narkis model which accounts for the adhesion between the dispersed phase and the matrix shows negative values, which indicates that there is significant adhesion between the phases in the blend.²⁹

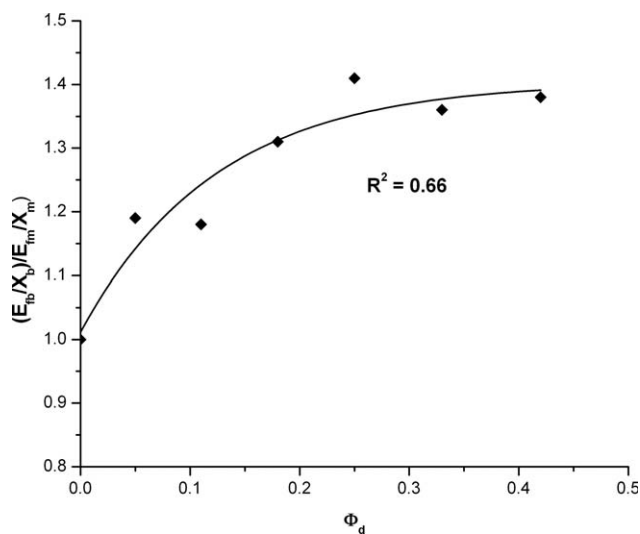


Figure 8 Dependence of normalized relative flexural moduli $[(E_{fb}/X_b)/(E_{fm}/X_m)]$ of PEEK/PEK blends (\blacklozenge) as a function of Φ_d .

To evaluate the effects of interphase adhesion and the sequential decrease in crystallinity of PEEK/PEK blends the normalized relative tensile strength $[(\sigma_{tb}/X_b)/(\sigma_{tm}/X_m)]$ was plotted versus Φ_d (Figure 5). The data were higher than unity increasing slowly up to $\Phi_d = 0.25$, which then almost leveled off. This happened because of the influence of overall crystallinity and crystalline shape in addition to the degree of compatibility of the blend system.¹³ Since the effect of decrease in crystallinity has been accounted for by dividing the crystallinity of the matrix, the value of the parameter higher than unity can be taken as the direct indication of phase interaction. The explanation regarding the overall crystallinity we have taken care in the previous section.

Elongation-at-break

Variations of relative strain-at-break ($\varepsilon_b/\varepsilon_m$) versus Φ_d are shown in Figure 6. The value increases with increase in Φ_d , at $\Phi_d = 0.33$ the value was ~ 1.16 times that of matrix. The data were compared with Nielsen's model for perfect adhesion³⁰ [eq. (6)]:

$$\varepsilon_b/\varepsilon_m = 1 - \Phi_d^{1/3} \quad (6)$$

The experimental data were higher than the model. In the two phase system the interphase characteristics control the blend performance. As the PEEK/PEK blend is miscible,¹³ increase in the elongation-at-break was observed.

Figure 7 exhibits the plot of normalized relative elongation-at-break $[(\varepsilon_b/X_b)/(\varepsilon_m/X_m)]$ versus Φ_d . It may be noted that up to $\Phi_d = 0.33$ the data increasing and the data above unity indicates that despite the interphase interaction and sequential decrease in

crystallinity the dispersed phase softens the matrix. The lower value of normalized relative elongation-at-break at $\Phi_d = 0.25$ indicates that at these PEK concentrations the matrix stiffening predominates to an extent compared with the softening effect.

Flexural properties

The flexural properties are presented as the ratio of the property of the blends (subscript b) to that of the PEEK matrix (subscript m) versus Φ_d of the PEK. Some of the data are presented in the normalized form by dividing the moduli with the crystallinity of the PEEK in the blend and the matrix $[(E_{fb}/X_b)/(E_{fm}/X_m)]$ and plotted as functions of Φ_d , in Figure 8.

The data were higher than unity increasing slowly up to $\Phi_d = 0.25$, which then leveled off with further increase in Φ_d with a correlation coefficient in terms of determination factor $R^2 = 0.66$. This implies that 66% of the changes in flexural modulus can be explained by the linear relationship of volume fraction of the PEK added in PEEK and the crystallinity of the blends. In Figure 9, dependence of normalized relative flexural strength $[(\sigma_{fb}/X_b)/(\sigma_{fm}/X_m)]$ of PEEK/PEK blends (\blacklozenge) as a function of Φ_d is shown with the value of $R^2 = 0.69$. The value of both flexural strength and modulus increased up to the volume fraction of 0.25 and then leveled off with further increase in Φ_d . This happened because both the components in the blend system were miscible.¹³

Fracture surface morphology and impact strength

The SEM photomicrographs of PEEK/PEK blends at varying Φ_d are shown in Figure 10(a–d). Micrographs show that the blends exhibit a homogeneous

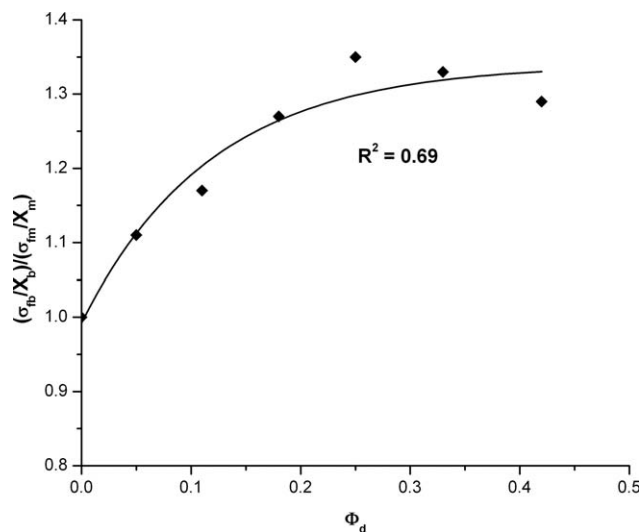


Figure 9 Dependence of normalized relative flexural strength $[(\sigma_{fb}/X_b)/(\sigma_{fm}/X_m)]$ of PEEK/PEK blends (\blacklozenge) as a function of Φ_d .

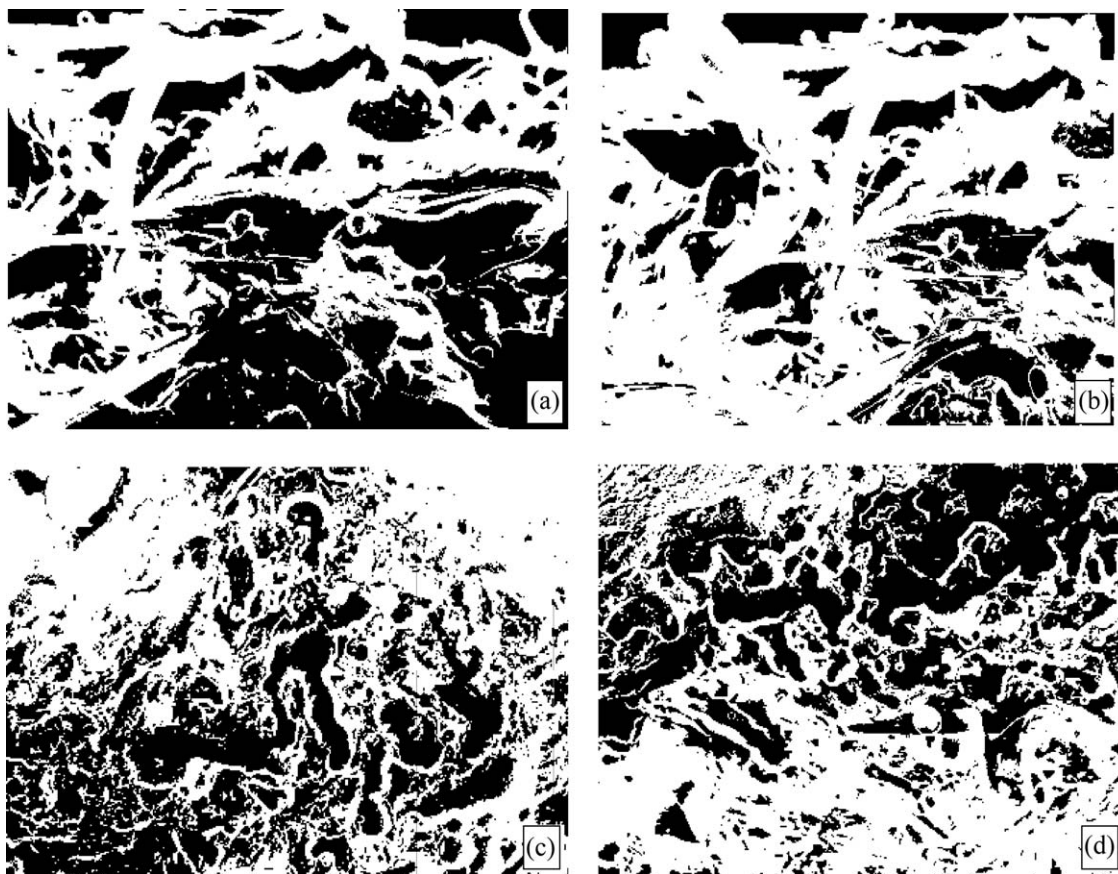


Figure 10 SEM photomicrographs of PEEK/PEK blends at varying Φ_d : (a) 0.11, (b) 0.18, (c) 0.33, and (d) 0.42.

structure, further indicating miscibility in this blend system. The miscibility of the blend is already reported elsewhere.¹³ For brevity, results of other blend compositions are not shown. In the SEM photograph, spherulites are clearly visible. With the increase of PEK concentration these spherulites formation have changed the morphology slightly. Otherwise, they all are similar to what is shown in Figure 10(a–d) indicating almost similar homogeneous structure free of any separated domains at the obtainable resolution of SEM.

The Izod impact strength (I_b) of the PEEK/PEK blends against the volume fraction (Φ_d) of PEK are given in the Table IV. The variations of normalized relative impact strength of the blends $[(I_b/X_b)/(I_m/X_m)]$

TABLE IV
Izod Impact Strength (I_b) Versus Φ_d in
PEEK/PEK Blends

Φ_d	I_b (J/m)
0	82.0
0.05	80.3
0.11	79.5
0.18	78.3
0.25	77.0
0.33	75.7
0.42	74.5

versus Φ_d , are shown in Figure 11. The parameter decreases marginally with Φ_d , the data follow a linear relationship with a value of $R^2 = 0.79$. This happened due to the degree of crystallinity as both the components in the blend system is semicrystalline.¹³ Crystallinity generally decreases the impact strength of polymers that have a T_g well above the

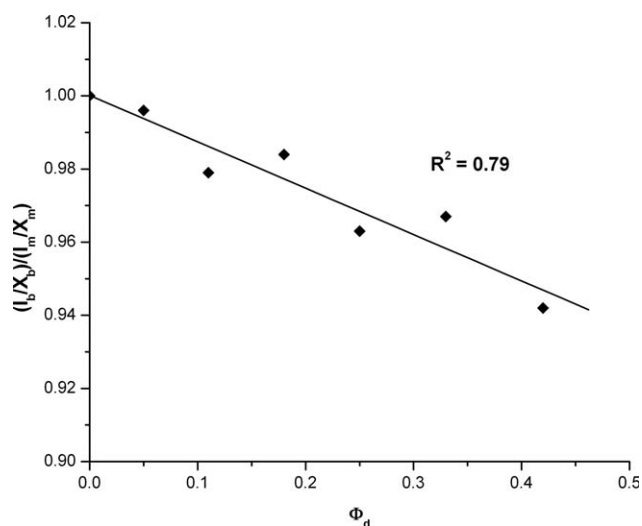


Figure 11 Plot of normalized relative impact strength $[(I_b/X_b)/(I_m/X_m)]$ of PEEK/PEK blends (\blacklozenge) versus Φ_d .

test temperature. As the spherulitic structure of such materials increases, as result of slow cooling from the melt or by annealing below melting point, the impact strength decreases.³⁰ The T_g value of PEK is higher than that of PEEK, so it does not favor the increase in impact strength. Hence, the marginal decrease in impact strength was observed.

CONCLUSIONS

Addition of PEK in PEEK enhances the tensile strength, flexural strength, flexural modulus, and elongation-at-break. The enhancement of the tensile strength, flexural strength, and modulus was due to the good adhesion of two polymers and the overall crystallinity. From the analysis of normalized tensile properties interphase adhesion and stress concentration factor were indicated. The increase in elongation-at-break was against the Nielsen's model [eq.(6)] due to the miscibility of the blends and semi-crystalline blend components. Tensile modulus decreased as the crystallinity of the blends decreased. Impact strength decreased as crystallinity generally decreases the impact strength of polymers that have a T_g well above the test temperature.

The authors thank the authorities of Defence Research and Development Organization for giving permission to publish the articles. The authors would also like to acknowledge the help of M/s Kabita Agarwal and Mr. Mahender Prasad for SEM studies.

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